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STRESS CORROSION CRACKING OF WROUGHT AND P/M HIGH
STRENGTH ALUMINUM ALLOY..(U) CARNEGIE-MELLON UNIV
PITTSBURGH PA DEPT OF METALLURGY AND MAT..

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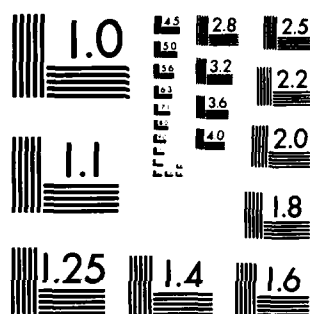
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We remain confident that we have established the basis and a good portion of the results necessary to understand, predict and model the role of hydrogen in stress corrosion cracking of high-strength aluminum alloys.

10 to 15 - 15 to 20 cm²/s

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Grant Number: AFOSR 81-0041
Principal Investigators: A. W. Thompson
I. M. Bernstein

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1.0 ABSTRACT OF RESULTS

The combined results of the first two years of the program are presented, with emphasis on the stress corrosion cracking and hydrogen embrittlement of the P/M X-7090 Al alloy. More complete results on 7075 are also given. In particular, the role of temper and loading mode and susceptibility were examined for three test methods---time to failure of notched round bar specimens in a brine solution; straining electrode tests on notched round specimens under cathodic charging; and tensile tests on hydrogen pre-charged notched round specimens. Another study examined the role of concurrent recovery processes during slow strain rate testing of cathodically charged 7075 and established that hydrogen outgassing is not a factor in the recovery, which instead is due to internal rearrangement of hydrogen to various trap sites. Also, a successful measurement of hydrogen diffusivity in aluminum has been made at room temperature using sandwich type specimens. The value obtained, about $10^{-15} \text{ cm}^2/\text{s}$ is one of the first successful measurements of this type.

We remain confident that we have established the basis and a good portion of the results necessary to understand, predict and model the role of hydrogen in stress corrosion cracking of high-strength aluminum alloys.

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MATTHEW J. KERPER
Chief, Technical Information Division

2.0 TECHNICAL RESULTS

The intent of the overall program is to identify the role played by hydrogen in stress corrosion cracking of high strength aluminum alloys first by establishing microstructural and fractographic correlations with cracking behavior and, then, to develop specific criteria for microstructural optimization in both powder and ingot alloys. Specific emphasis has been placed on the role of such metallurgical variables as precipitate type, size, and distribution, in order to develop them as central variables for the production of wrought and powder high-strength aluminum alloys, most notably X-7090 and IM 7075, more resistant to hydrogen embrittlement (HE) and/or stress corrosion cracking (SCC).

Second-year results are summarized in the following sections and will cover the following specific topics: The role of hydrogen microstructure and loading on the stress corrosion cracking response of X-7090 and 7075; hydrogen recovery studies in 7075; hydrogen diffusivity and permeability in aluminum.

2.1 The Role of Hydrogen in the Stress Corrosion Cracking of P/M X-7090 Al

A general description of P/M X-7090 was provided previously⁽¹⁾, covering microstructural aspects as well as its preliminary response to stress corrosion cracking and hydrogen embrittlement. What follows is more detailed results summarizing both the first and second year.

A slip characterization study was undertaken in an attempt to determine whether slip mode could be altered in X-7090 and thus help explain the SCC response for the various aged conditions. TEM foils from under-aged (100°C - 24 hr.) as well as room temperature aged (48 hr.) specimens showed only wavy or homogeneous slip. It would appear that the fine dispersion of Co_2Al_9 and oxide particles may have more control of slip mode in this alloy than do the aging precipitates; thus, the importance of slip planarity is yet to be demonstrated in this alloy.

Environmental mechanical testing included three test methods, all employing the loading mode technique, which can help discriminate between the relative contributions of hydrogen embrittlement and anodic dissolution to the overall SCC behavior of ferrous and nonferrous alloys⁽²⁾. The primary test method to establish general response was the use of

constant load (or constant torque) applied to a notched round bar held at the free corrosion potential in a chromate-inhibited brine solution⁽³⁾. Although 3-1/2% NaCl solutions were used satisfactorily for previously conducted tests on 7075, we found extreme exfoliation-type damage on torsion X-7090 specimens tested in 3-1/2% NaCl. The exfoliation plates appear to be aligned with oxide stringers in the P/M alloy. The chromate-inhibited brine solution eliminated this damage. This difference in exfoliation behavior is worthy of additional study because of its potentially deleterious impact on service life under specific loading conditions.

Time-to-failure results for the underaged condition for X-7090 are shown in Fig. 1, with stress intensity values normalized to their air test values. The separation between the two loading mode curves suggests that hydrogen plays a role in the SCC of underaged X-7090⁽²⁾. Figure 2 shows results for peak aged X-7090. The curves tend to be spread apart, particularly at lower applied stress intensities, again suggesting a hydrogen contribution. Fig. 3 shows results for overaged X-7090. This graph depicts a highly unusual, unexpected result that the Mode III curve now lies below the Mode I curve, implying that specimens fail sooner in torsion than in tension at equivalent initial normalized loadings. At this stage, subject perhaps to reinterpretation, it appears that in overaged X-7090 Al, the SCC process is more likely controlled by an anodic dissolution mechanism rather than hydrogen embrittlement. Test results for notched tension specimens tested in 3-1/2% NaCl (saltwater) are included in Figure 2 for comparison to the results in chromate-inhibited brine solution. The chromate-inhibited brine solution appears to be more aggressive than the 3-1/2% NaCl solution for peak aged X-7090. Note that torsion test results are not shown for 3-1/2% NaCl solutions because of the previously mentioned exfoliation damage.

Figure 4 combines the Mode I data of Figures 1-3 for X-7090. Increasing the degree of aging (from underaged to peak to overaged) increases the resistance to SCC, as has been previously demonstrated for 7075⁽⁴⁾. On the other hand, the comparison summary of Mode III results, shown in Figure 5, emphasizes the previously discussed anomaly for the overaged treatment, where now in torsion, it appears to be the most susceptible condition. Confirmation of these results is required since the small differences between the curves in Figure 5 may simply imply that the SCC behavior in Mode III is the same for all three tempers of X-7090.

The second test method used is the straining electrode test where notched round specimens were cathodically charged under a constant potential of -1500 mV versus the SCE in hydrochloric acid (pH 1), while being strained to failure in either Mode I or Mode III. The value of this approach is that hydrogen can be introduced under more severe entry conditions than with constant load or other tests, allowing maximum effects to be observed and studied. Results are shown in Figure 6 with again the stress intensities normalized to their air test values. In addition to the three tempers used in the constant load tests, the SET testing also included specimens aged at room temperature for four days, which produced a microstructure with a yield strength about 30% lower than that of the peak aged condition⁽¹⁾. Because each bar in Figure 6 represents a single specimen, statistical confidence limits are not included, but a trend is evident of a decreasing difference between Mode III and Mode I sensitivity as aging is increased. This result coincides with the constant load data, even to the point of a reversal between Mode I and Mode III for the overaged temper, providing additional confidence as to the validity of the overaged data.

A third test technique, hydrogen precharging, a less severe test than the SET, was also used. Notched round specimens were cathodically precharged for 10 hours in pH 1 hydrochloric acid at -1500 mV SCE and were strained to failure at crosshead rates of 0.2 mm/min. (Mode I) or 0.001 rpm (Mode III). The results of these tests are similar to those of the SET tests, but the Mode I/Mode III differences are expectedly smaller, since the precharge conditions are not as severe as those of the SET.

All failed specimens from the three test series have been examined fractographically in the SEM. Thus far, no dramatic trends in fracture mode, which can correlate with the large differences in response, have been found. Figure 7 shows typical fracture surfaces for torsion as well as tension specimens. Both modes show a dimpled response with the tensile specimen having a more intergranular dimpled appearance. Figure 8 shows a typical SET specimen with secondary cracks aligned in the direction of the oxide stringers. The cracking in the right photomicrograph of Figure 8 is transgranular with local regions of tearing. No intergranular cracking has been observed in these studies, even near the surface in contrast to the 7075 results⁽⁵⁾.

Double cantilever beam specimens are also being tested to evaluate the role of temper condition on crack initiation and velocity in X-7090. Because

the X-7090 plate made available to us was thinner than desired, (11 mm vs. 25.4 mm) for a conventional fracture mechanics specimen, stiffener arms were attached above and below the crack plane to produce a more valid DCB with dimensions of 25.4 x 25.4 x 114.3 mm and with a S-L orientation. While arm break-off was frequently encountered, probably because of the unusual specimen design, a V-K curve for peak aged X-7090 was generated; both underaged and overaged specimens are currently in testing. In addition, a 7075-T6 specimen has been modified with stiffener arms so the X-7090 results can be compared with results on 7075 in case the modified DCB results cannot be directly compared with conventional DCB results.

2.2 Stress Corrosion Cracking of 7075 Al - Mode I/Mode III Testing

We have also been supplementing previous SCC test results on 7075 Al⁽⁵⁾, with constant torque tests to facilitate comparison with the Mode I notched longitudinal test results, in a like manner to the X-7090. These tests were, similar to the Mode I specimens, conducted in a 50°C, 3.5% NaCl solution. Time-to-failure results are shown in Figures 9, 10, and 11 for the underaged (100°C - 24 hr.), peak aged (120°C - 24 hr.) and the overaged (160°C - 24 hr.) conditions, respectively. Since the Mode I loading caused shorter failure times than Mode III for the underaged and peak aged conditions, a hydrogen contribution to the SCC is again suggested. For the overaged condition, however, Mode III again had shorter failure times than Mode I, following similar behavior for overaged X-7090. As was stated for X-7090, the overaged condition of 7075 appears to stress corrosion crack with little or no hydrogen contribution.

Additional constant load/torque tests are being completed on notched short transverse 7075 specimens, a more susceptible orientation, to help assess the role of grain orientation on SCC behavior. The test conditions are the same as for the longitudinal specimens. While Mode III results are incomplete, Mode I results are shown in Figure 12, which shows little difference in failure times between underaged and peak aged specimens. The overaged condition is much more resistant to cracking, as was found previously for longitudinal specimens⁽⁵⁾.

2.3 Conclusions and Additional Work

- For X-7090 as well as for 7075, SCC resistance increases as aging proceeds from underaged to peak aged to overaged. This has been demonstrated in both constant load and SET testing and in a number of aqueous environments with very different pH's.
- For both X-7090 and 7075, the underaged and peak aged conditions show a strong hydrogen contribution to SCC. Overaged conditions of both alloys show a possible reversal in Mode I and Mode III, at least suggesting a more limited role of hydrogen in SCC of overaged alloys.

As inferred from the above presentation and discussion of results, additional work is necessary to gain a more complete understanding of the role of hydrogen in the SCC of high strength 7075 and X-7090 aluminum alloys.

While the fractography seems to be dominated by constituent particles such as oxides, the trends in failure times in the SCC tests indicate a role that must be played by the aging precipitates, but not apparent from fractography. For that reason, we believe it useful to characterize carefully the aging sequence of the microstructure via TEM. If the fine grain size impedes precipitate identification, the X-7090 will be treated to give a larger grain size prior to solution treating and aging. This larger grained material may also be used as part of a study to characterize the slip mode as a function of age condition in X-7090. Previous work in the group has established the processing conditions necessary to achieve the desired grain growth.

The testing of modified DCB specimens will be completed to evaluate the role of microstructure on crack initiation and propagation in X-7090 and compared to 7075. Such information will be useful in understanding the SET and constant load/torque test results. Constant torque tests will be completed on 7075 specimens in the short transverse orientation to provide insight into the role of grain orientation on SCC.

An ongoing analytical effort continues that involves determining the most appropriate and accurate way to compare Mode I and Mode III results for sharply notched round specimens. Greater confidence is needed to ensure that the observed Mode I/Mode III reversal is a real effect and not simply an arti-

fact of how the normalized stress intensities are compared. An investigation of this problem has begun and it appears that an alternate method of comparison may be warranted since the method used for calculating stress intensities for Mode III specimens is incorrect at least for uncracked specimens. However, we remain convinced as to the absolute value of Mode I/Mode III comparisons as well as to the general trends observed, particularly since it appears that a more correct analysis will widen observed differences between Mode I and Mode III.

2.4 Recovery Behavior of Hydrogen Charged 7075

Recent work on hydrogen embrittlement of a high strength Al alloy as a function of strain rate has reiterated the reversibility of the embrittling effect of hydrogen; below a certain "critical strain rate" which can depend on alloy content and microstructure, the ductility of embrittled materials increases. We wish to establish if the observed recovery in ductility at slow strain rates is due either to hydrogen loss from the specimen or to trapping of hydrogen within the material at sites not involved in the ductile fracture process. The former possibility, hydrogen loss, has been discounted⁽⁶⁾ in carefully-conducted tests; the latter possibility remains to be investigated.

Another point needing clarification is the use of laboratory air, approximate relative humidity 60%, as a reference environment.

Tensile tests of uncharged 7075 specimens of different tempers were performed at various initial strain rates ranging from 3.33×10^{-5} to 3.33×10^{-3} /s in a vacuum chamber at a pressure of 2×10^{-6} torr, to prevent any hydrogen embrittlement due to moisture. Charged specimens in 0.1 N HCl at a potential of -1500 mV vs. SCE for 10 hrs. at RT were either tested in the same vacuum chamber or in a dry hydrogen atmosphere having a pressure slightly over 1 atm. The results obtained are summarized in Figure 13 where the ductility of uncharged specimens is seen to increase monotonically with decreasing strain rate, as is usually observed, increasing at about 2.8% per order of magnitude in strain rate. The test results of the charged specimens show the same dependence of ductility on the applied strain rate as found previously⁽⁶⁾. Maximum embrittlement (lowest RA) is reached at a strain rate of about $3 \times 10^{-4} \text{ s}^{-1}$. Further, no systematic difference in ductility can be determined between the tests of charged specimens conducted in vacuum and in dry hydrogen

gas. To ensure that the time in vacuum during the evacuation of the chamber to $< 2 \times 10$ torr had no influence on the recovery behavior, two specimens were kept in vacuum for 48 hrs. before testing. The results shown in Figure 13 (data points marked with an arrow) indicate that this treatment did not influence the ductility.

Examining the fracture surfaces of specimens tested in vacuum and in dry hydrogen gas revealed no significant difference in fracture mode or overall fracture appearance.

Thus, the recovery behavior of the tested 7075-T6 alloy is not influenced by the hydrogen pressure of the surrounding environment, up to 1 atm pressure. It also seems probable that recovery of ductility at room temperature is associated with hydrogen transport by dislocations because storage of charged specimens in vacuum prior to testing has no influence on ductility. Similar observations have been made before⁽⁶⁾. Therefore it is suggested that recovery of ductility at slow strain rates is associated with hydrogen trapping within the material. Hydrogen loss out of the specimen is considered to become important only at elevated temperatures, when lattice diffusion is enhanced, or at very long times.

However, these results, that internal rearrangement of hydrogen to innocuous traps can account for the recovery of ductility at low strain rates, can only be valid under conditions of fixed and moderate content of hydrogen. When the content is large, or the external supply unlimited, it would be expected theoretically⁽⁷⁾ and experimentally⁽⁸⁾ that innocuous traps would no longer act to reduce embrittlement at slow strain rates. This is exactly what has been observed for high-strength Al alloys; for extensive charging under high fugacities or for testing in environments which can supply hydrogen, no recovery at low strain rates was observed. Thus, the behavior shown in Figure 13 of a critical strain rate for maximum embrittlement should only be observed when hydrogen content is fixed and small enough to permit significant internal trapping at innocuous sites, prior to their being saturated.

To summarize this study, which is being submitted for publication, the following conclusions have been reached:

- 1) The ductility decrease observed for specimens tested in laboratory air at slow strain rates can be due to hydrogen embrittlement by

moist air and does not occur if tests are conducted in vacuum or dried air.

- 2) Recovery of ductility below a certain strain rate is not influenced by low hydrogen pressure in the environment.
- 3) It is suggested that recovery is due to internal rearrangement of hydrogen at Cr-rich inclusions and at rather large particles, like η and T-phase precipitates in 7075-T6 and 7075-T73 alloys, which act as innocuous traps. This behavior should only be evident when testing under conditions of fixed and moderate hydrogen content.
- 4) The difference in recovery behavior between the tempers UT, T6 and T73 observed earlier is attributed to the presence of and the size of η and T-particles reducing by different amounts the hydrogen available to embrittle the material.

2.5 Hydrogen Diffusivity Through Aluminum

A critically needed piece of information is the permeability and diffusivity of hydrogen through aluminum at temperatures of interest to embrittlement effects, around room temperature. Because of its very low solubility and diffusivity in Al, researchers have relied on high temperature extrapolations, a very risky venture since trapping at lower temperature invariably leads to a much lower effective diffusivity than the extrapolated value. Knowledge of this type is absolutely essential in any successful model development of hydrogen effects in Al-based alloys.

We have recently developed a technique that has allowed us to successfully measure hydrogen diffusivity in pure Al. The key is the use of a sandwich specimen whereby aluminum and palladium are vapor deposited onto a pure iron substrate making up the cathodic side of the specimen. On the anodic side, palladium is electrodeposited onto the other side of the iron substrate to produce a surface with reproducible electrochemical properties. A schematic of the sandwich permeation specimen is shown in Figure 14. Permeation transients were obtained and analyzed to separate out the contributions of the iron and the interface to the overall rate. A room temperature diffusivity for the fine grained ($\sim .4\mu\text{m}$) vapor deposited aluminum of 10^{-15} to $10^{-16} \text{ cm}^2/\text{s}$ was obtained. The large difference between this and the value

of $\sim 10^{-9} \text{ cm}^2/\text{s}$ obtained from high temperature extrapolation has been rationalized as resulting from the low hydrogen solubility and high trap density of the specimen.

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4.0 AFOSR-SPONSORED PUBLICATIONS (1982)

1. R. E. Swanson, I. M. Bernstein, and A. W. Thompson, "Stress Corrosion Cracking of 7075 Aluminum in the T6-RR Temper," Scripta Met., Vol. 16 (1982), pp. 321-24.
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4. A. W. Thompson and I. M. Bernstein, "Quantitative Metallography and Fractography in Hydrogen Embrittlement Studies," in Advanced Techniques for the Characterization of Hydrogen in Metals (N. Fiore and B. J. Berkowitz, eds.) TMS, Warrendale, PA (1982), pp. 43-60.
5. A. W. Thompson and I. M. Bernstein, "Microstructural Effects on Hydrogen Embrittlement," in Hydrogen and Materials (P. Azou, ed.) Paris (1982), pp. 845-50.
6. A. W. Thompson and I. M. Bernstein, "Loading Mode (Mode I-Mode III) Testing for Stress Corrosion Cracking," in Environment-Sensitive Fracture: Evaluation and Comparison (E. N. Pugh and G. M. Ugiansky, eds.) ASTM, in press.
7. D. A. Hardwick, A. W. Thompson and I. M. Bernstein, "Effect of Copper Content and Microstructure on Hydrogen Embrittlement of Al-6Zn-2Mg Alloys," submitted to Met. Trans.
8. I. M. Bernstein and A. W. Thompson, "The Role of Microstructure in Hydrogen Embrittlement," A. R. Troiano Honorary Volume (R. Gibala, ed.), Academic Press, N. Y., in press.
9. I. M. Bernstein and A. W. Thompson, "The Importance of Transient Effects Resulting from Dislocation Transport of Hydrogen," in Atomistics of Fracture (R. M. Latanision, ed.), Plenum Press, N. Y., in press.

5.0 INTERACTIONS

5.1 Presentations

1. R. E. Swanson, I. M. Bernstein and A. W. Thompson, "Environmental Embrittlement of a High-Strength P/M Al Alloy," Symposium on High Performance Aluminum Powder Metallurgy Alloys Annual Meeting, AIME, Dallas, TX, 18 February 1982.
2. I. M. Bernstein and A. W. Thompson, "Microstructure, Trapping and Fracture Effects on Environmental Embrittlement," (invited), Staff Seminar, Martin Marietta Laboratories, Baltimore, MD, 8 March 1982.
3. A. W. Thompson and I. M. Bernstein, "Loading Mode (Mode I-Mode III) Testing for Stress Corrosion Cracking," ASTM Conference on Environment-Sensitive Fracture Evaluation and Comparison of Test Methods, National Bureau of Standards, Gaithersburg, MD, 26 April 1982.
4. I. M. Bernstein, "Material Constraints for High Technology Applications," Westinghouse Executive Program: Carnegie-Mellon University, Pittsburgh, PA, March 1982.
5. A. W. Thompson, "The Interaction of Metallurgy and Mechanic Viewpoints on Fracture," (invited), Seminar Series, Dept. of Mechanical and Environmental Engineering, University of California, Santa Barbara, CA, 17 May 1982.

6. A. W. Thompson, "The Hydrogen Economy - Materials Problems and Current Status," (invited) Spring Meeting, Pittsburgh Section, The Electrochemical Society, Monroeville, PA, 21 May 1982.
7. A. W. Thompson and I. M. Bernstein, "Microstructural Effects on Hydrogen Embrittlement," 3rd International Congress on Hydrogen and Materials, Paris, France, 8 June 1982.
8. R. E. Swanson, I. M. Bernstein and A. W. Thompson, "The Role of Hydrogen in the Stress Corrosion Cracking of X-7090 P/M Aluminum," Fall Meeting, TMS-AIME, St. Louis, MO, 27 October 1982.
9. I. M. Bernstein, "Microstructural Control of Mechanical Properties," Pittsburgh Night Lecture of the American Society for Metals, 18 November 1982.
10. I. M. Bernstein, "The Dependence of Properties on Microstructure," invited seminar, Lawrence Livermore Laboratories, Livermore, CA, 22 November 1982.

5.2 Technical Contacts with Other Investigators

The principal investigators continue to have significant and often extensive contacts with other AFOSR investigators in related fields, as briefly summarized below.

Professor J. L. Swedlow (solid mechanics) - Carnegie-Mellon University: Discussions and guidance for the development of finite element methods to study and model stress and strain localization.

Professor J. C. Williams - Carnegie-Mellon University: A continuing dialogue on the role of microstructures on strengthening mechanisms in aluminum alloys.

Professor R. P. Wei - Lehigh University: Discussions on the role of loading mode on discriminating fracture mechanisms.

Professor E. J. Starke - Georgia Institute of Technology: Discussions of the role of Cu additions to Al-Zn-Mg alloys on microstructure and SCC resistance.

5.3 Personnel

A. W. Thompson - Professor and Co-Principal Investigator
(18% AY, 28% summer).

I. M. Bernstein - Professor and Co-Principal Investigator
(7% AY, 16% summer).

R. E. Swanson - Graduate Student.

M. Mueller - Post-doctoral Associate

W. Y. Choo - Post-doctoral Associate (no cost)

J. Price - Undergraduate Student (no cost)

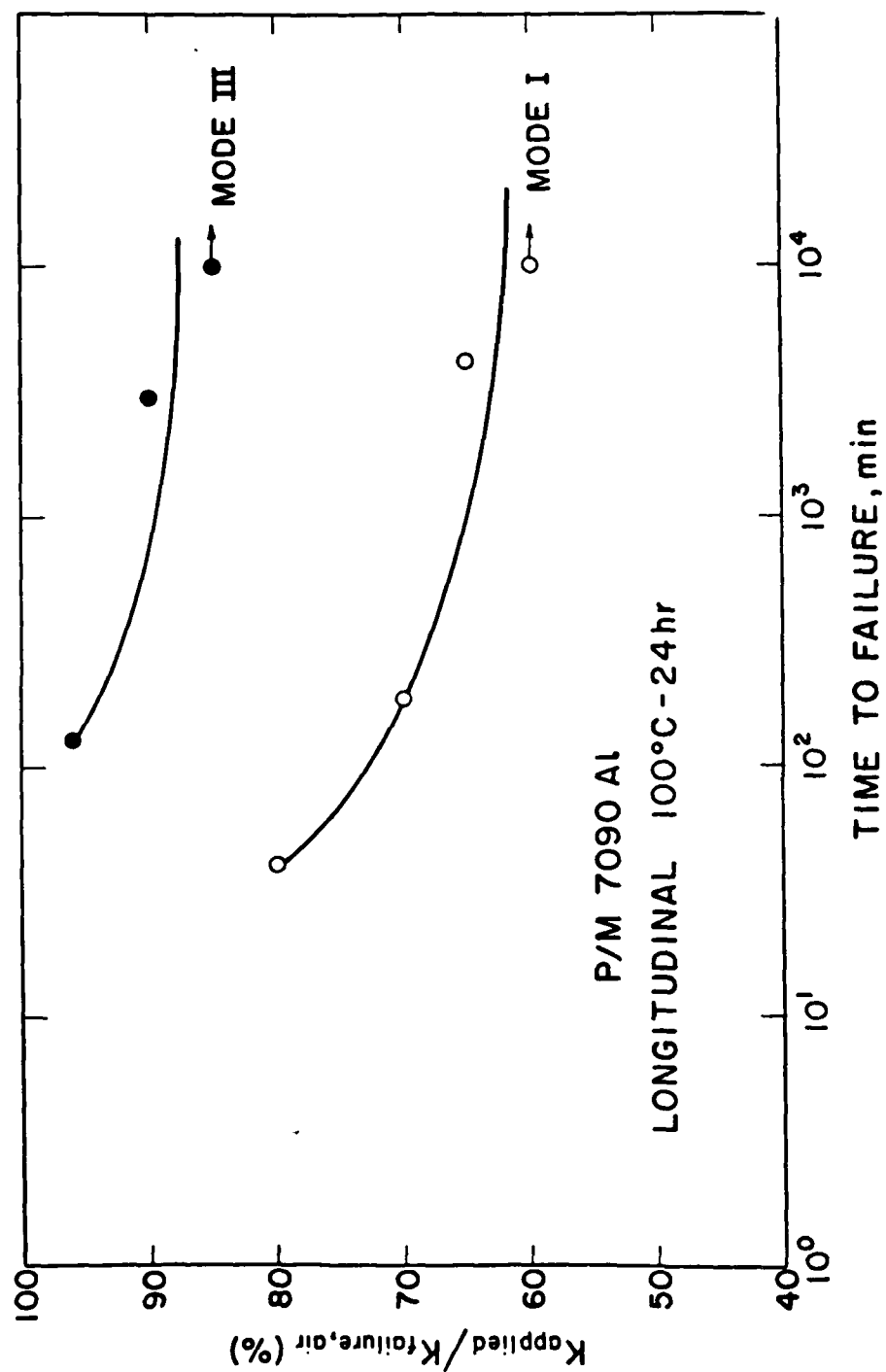


Figure 1 Constant Load/Torque SCC Results for Underaged P/M 7090 Al

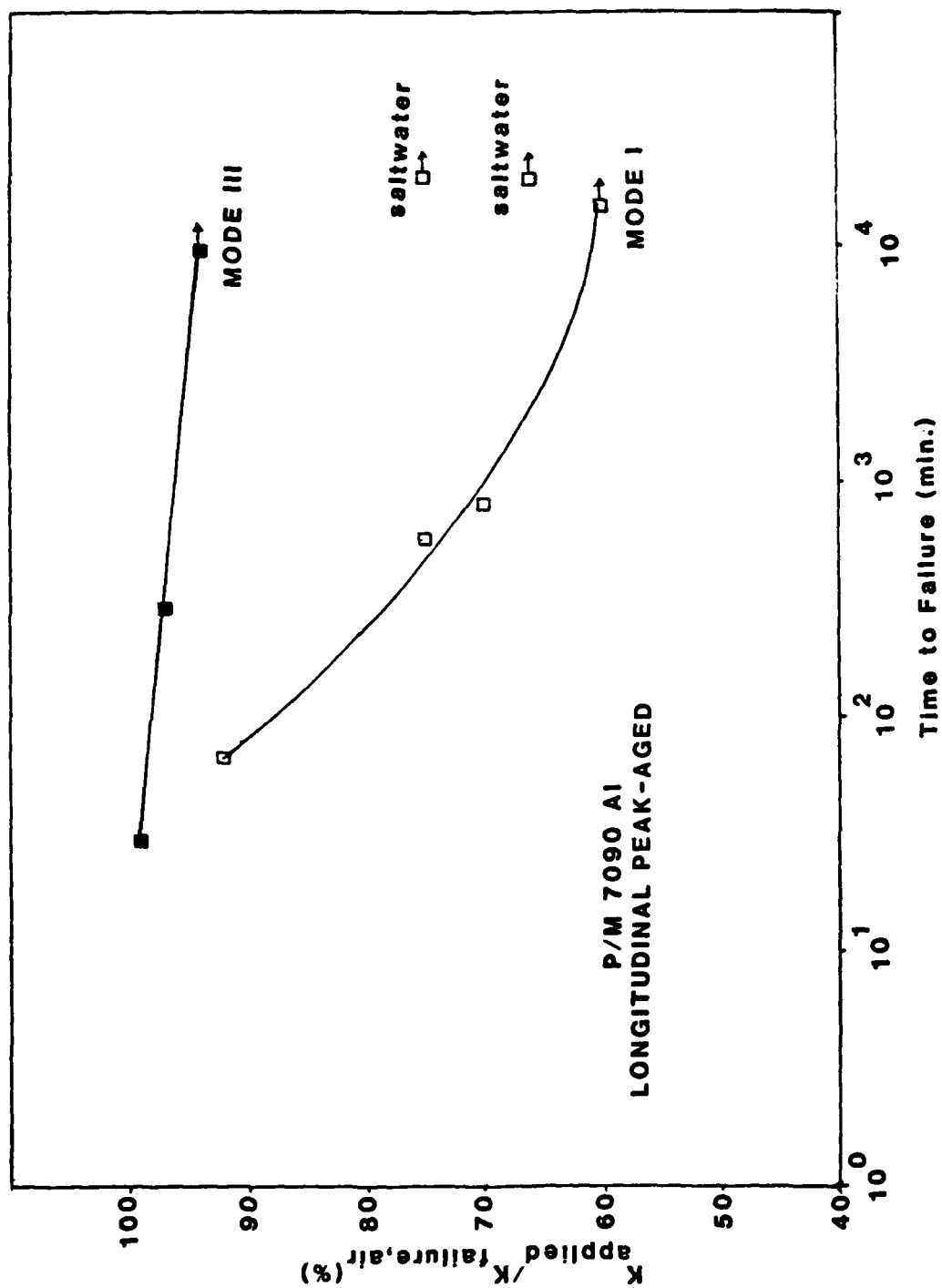


Figure 2 Constant Load/Torque SCC Results for Peak-Aged P/M 7090 Al

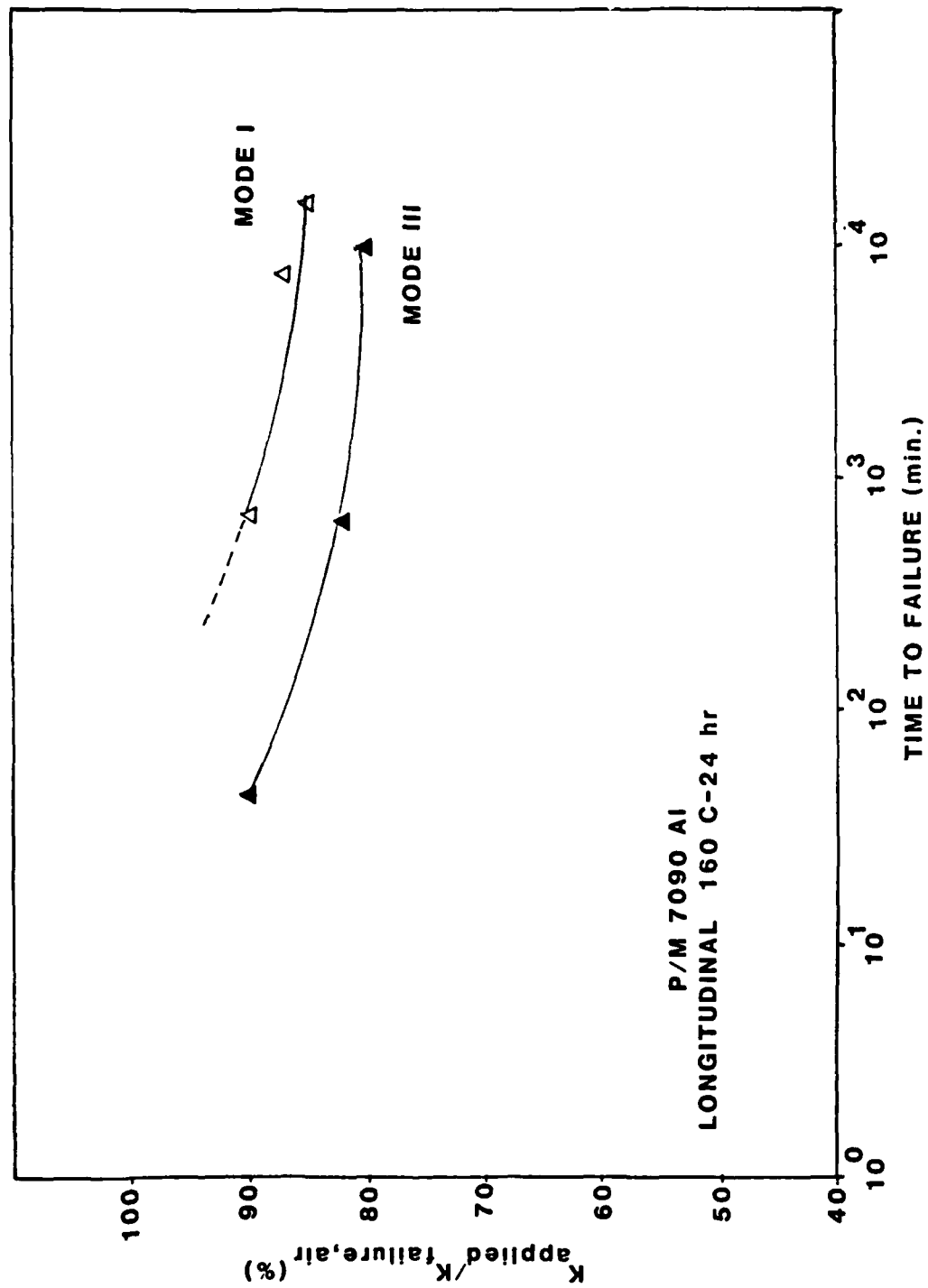


Figure 3 Constant Load/Torque SCC Results for Overaged P/M 7090 Al

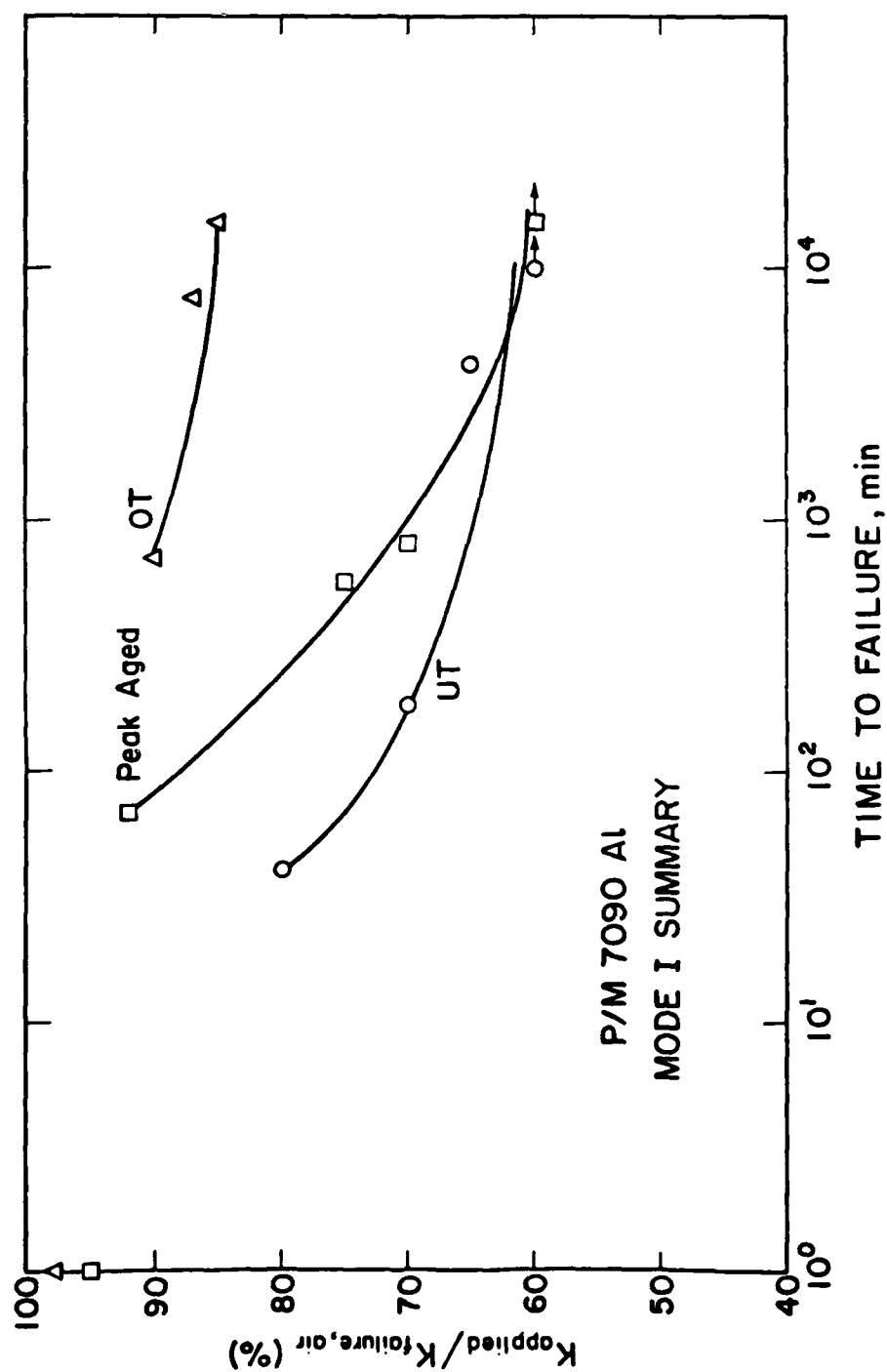


Figure 4 Constant Load SCC Summary for P/M 7090 Al

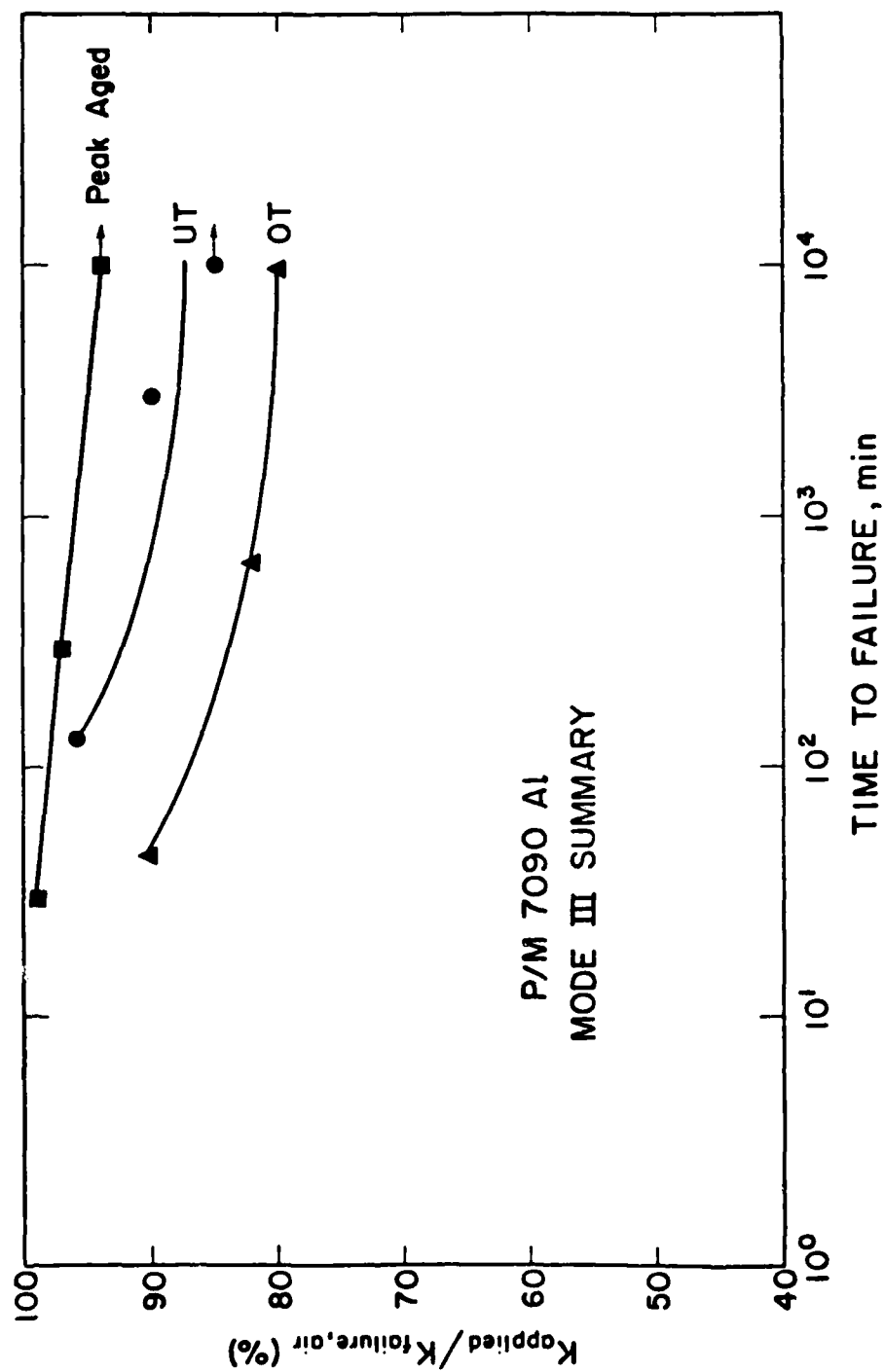


Figure 5 Constant Torque SCC Summary for P/M 7090 Al

SET LOADING MODE RESULTS

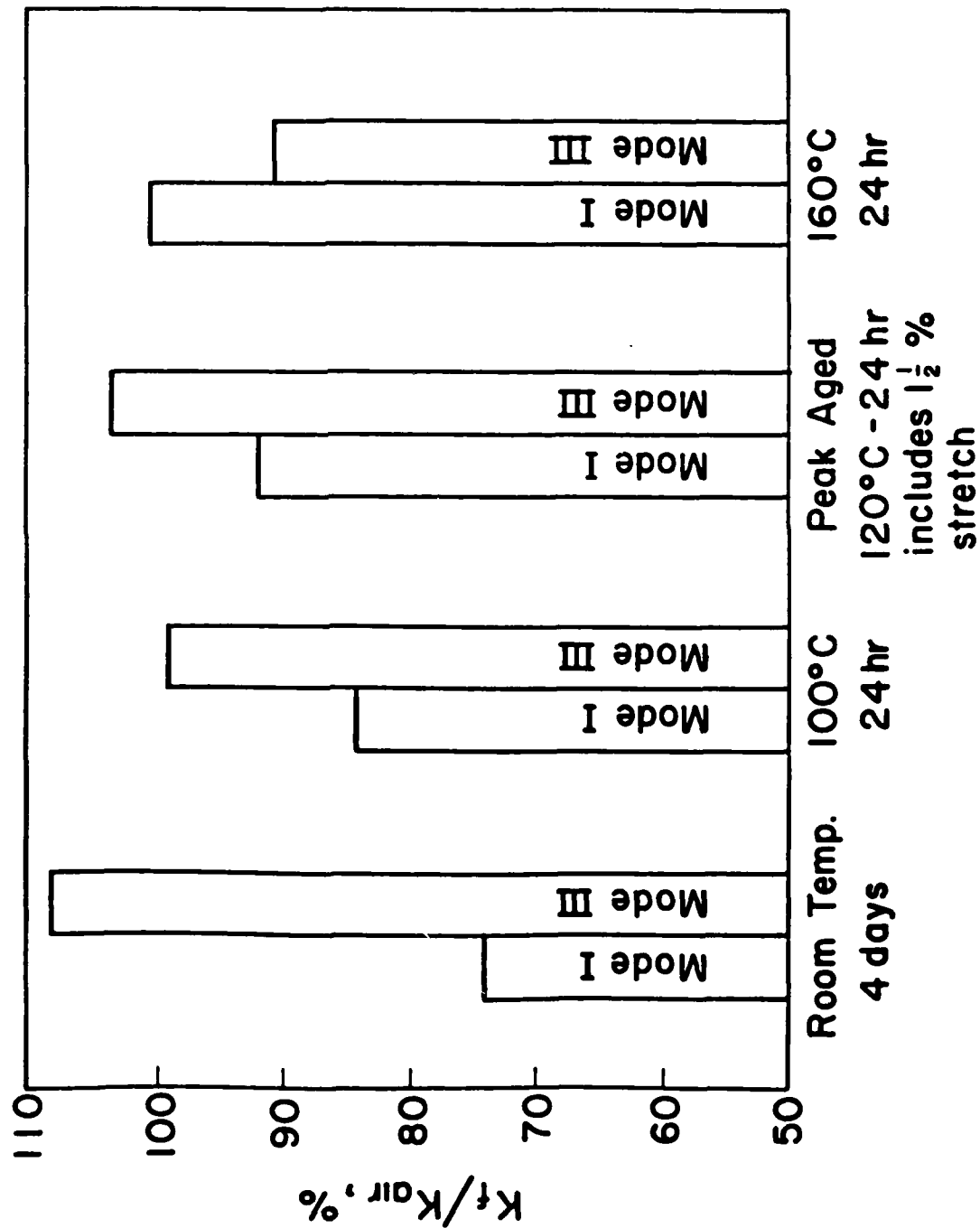
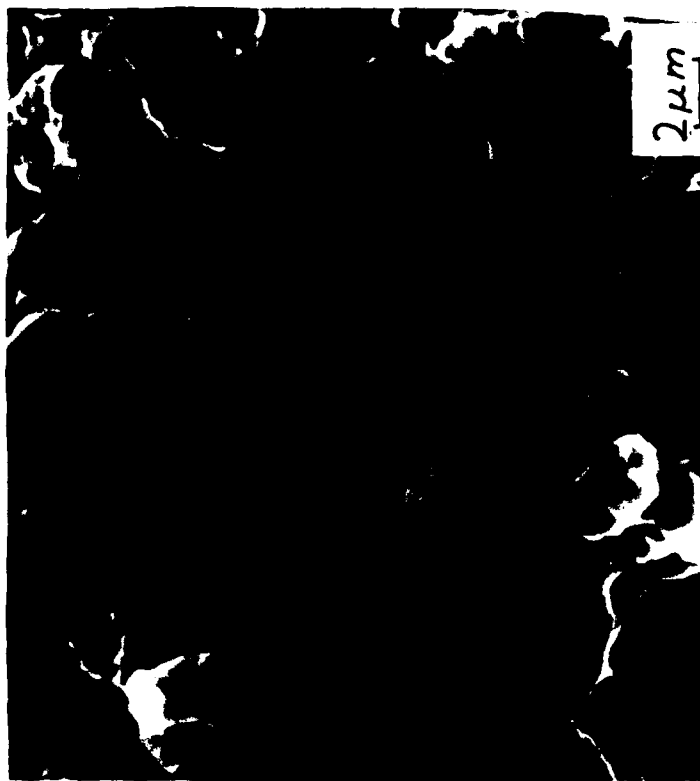


Figure 6

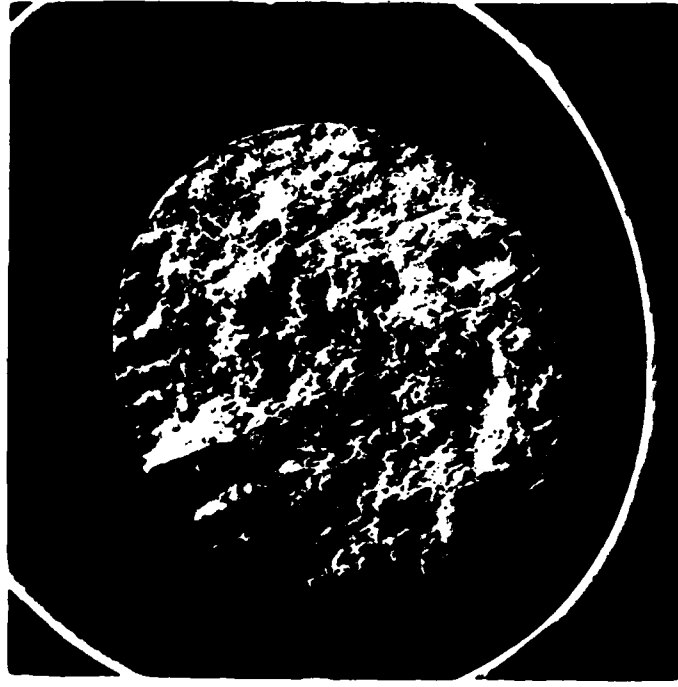


222 X-7090 Torsion Longitudinal
100°C - 24 hr. Constant torque - Inhibited brine



216 X-7090 Tension Longitudinal
Peak aged. Constant load - Inhibited brine

Figure 7



LONGITUDINAL TENSILE SPECIMENS

Underaged Temper
Straining Electrode Tests
pH 1 HCl
Cathodic Overpotential 150mV

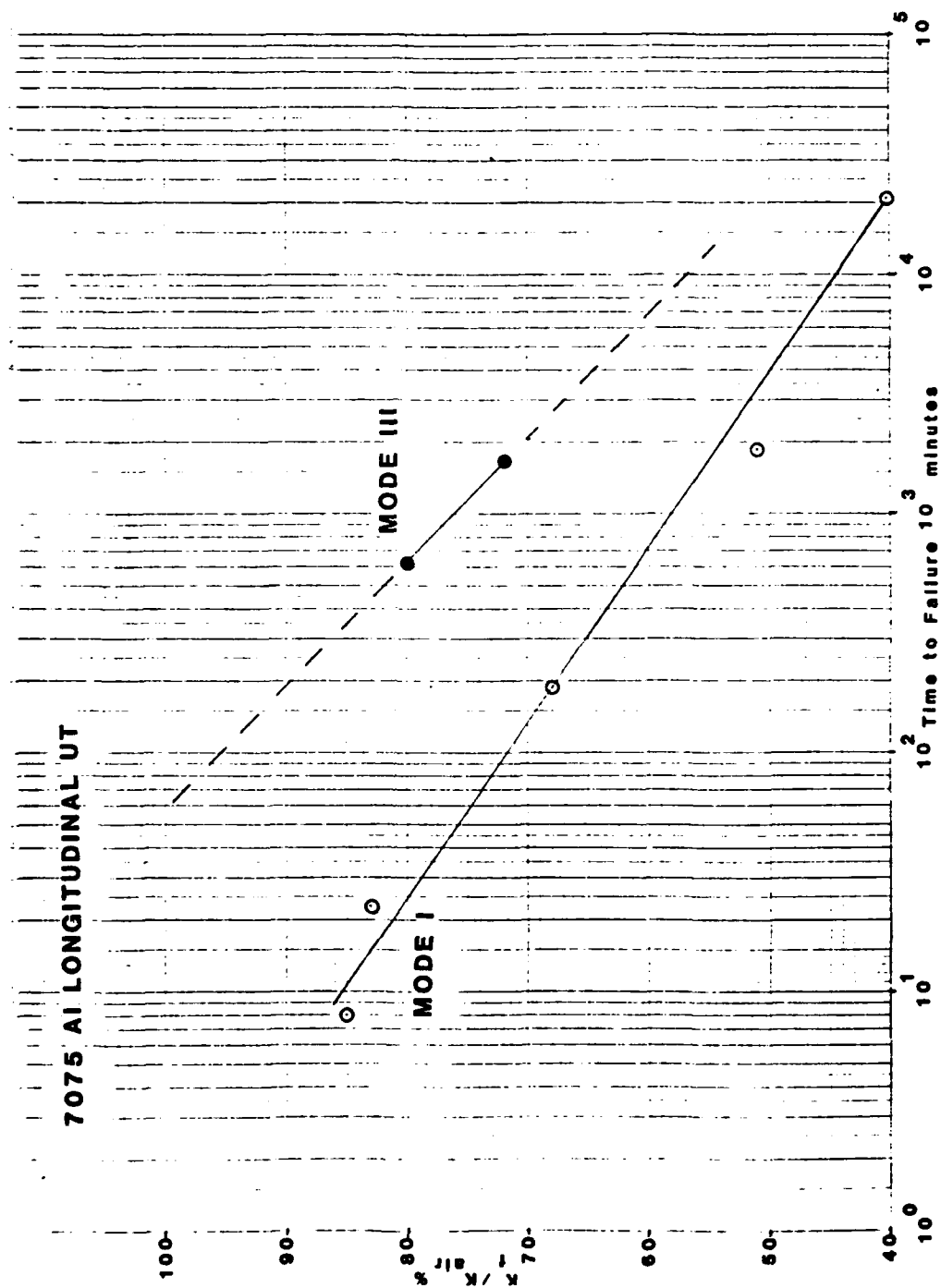


Figure 9 Constant Load/Torque SCC Results for Underaged 7075 Al - Longitudinal

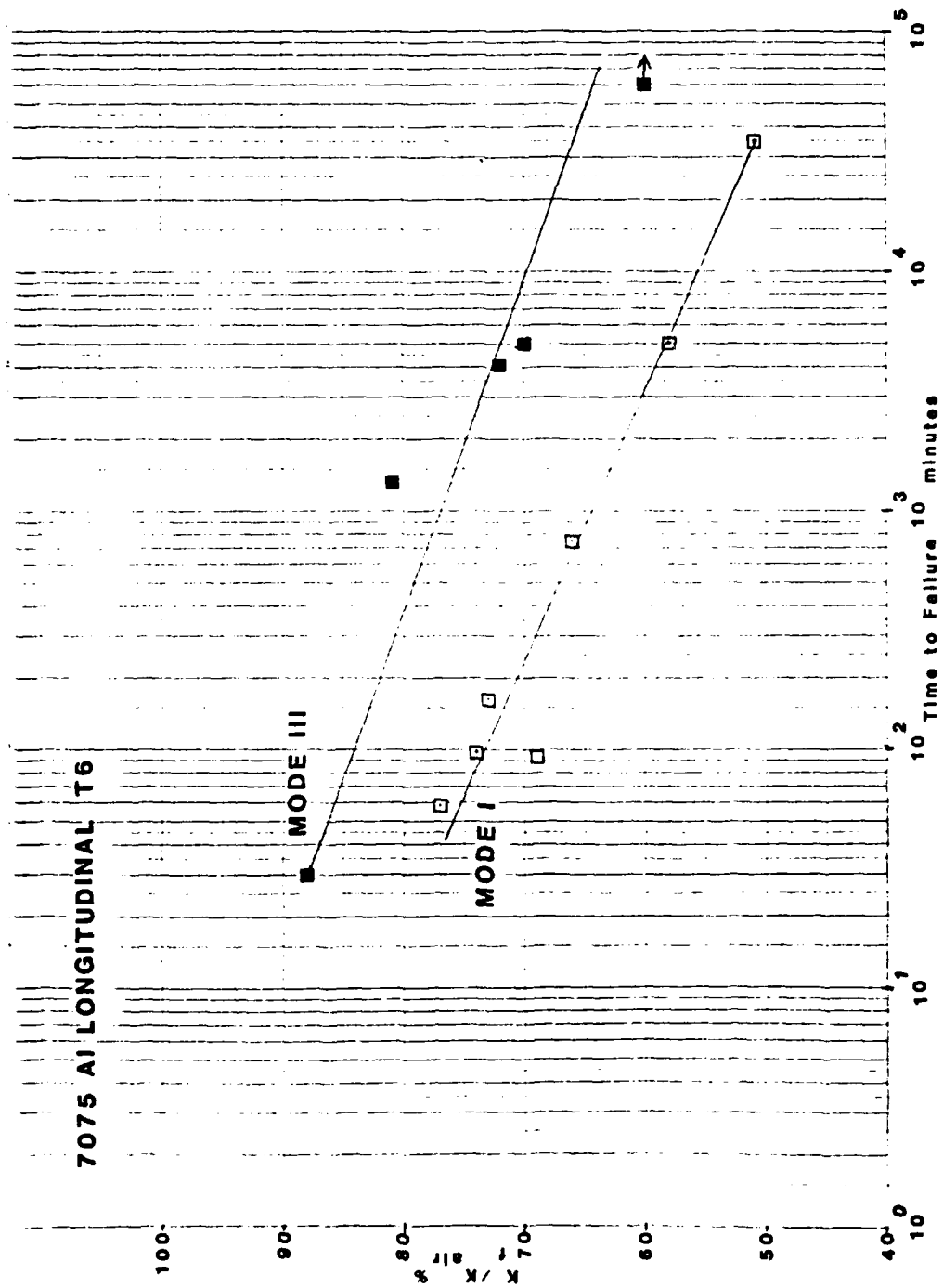


Figure 10 Constant Load/Torque SCC Results for Peak-Aged 7075 Al - Longitudinal

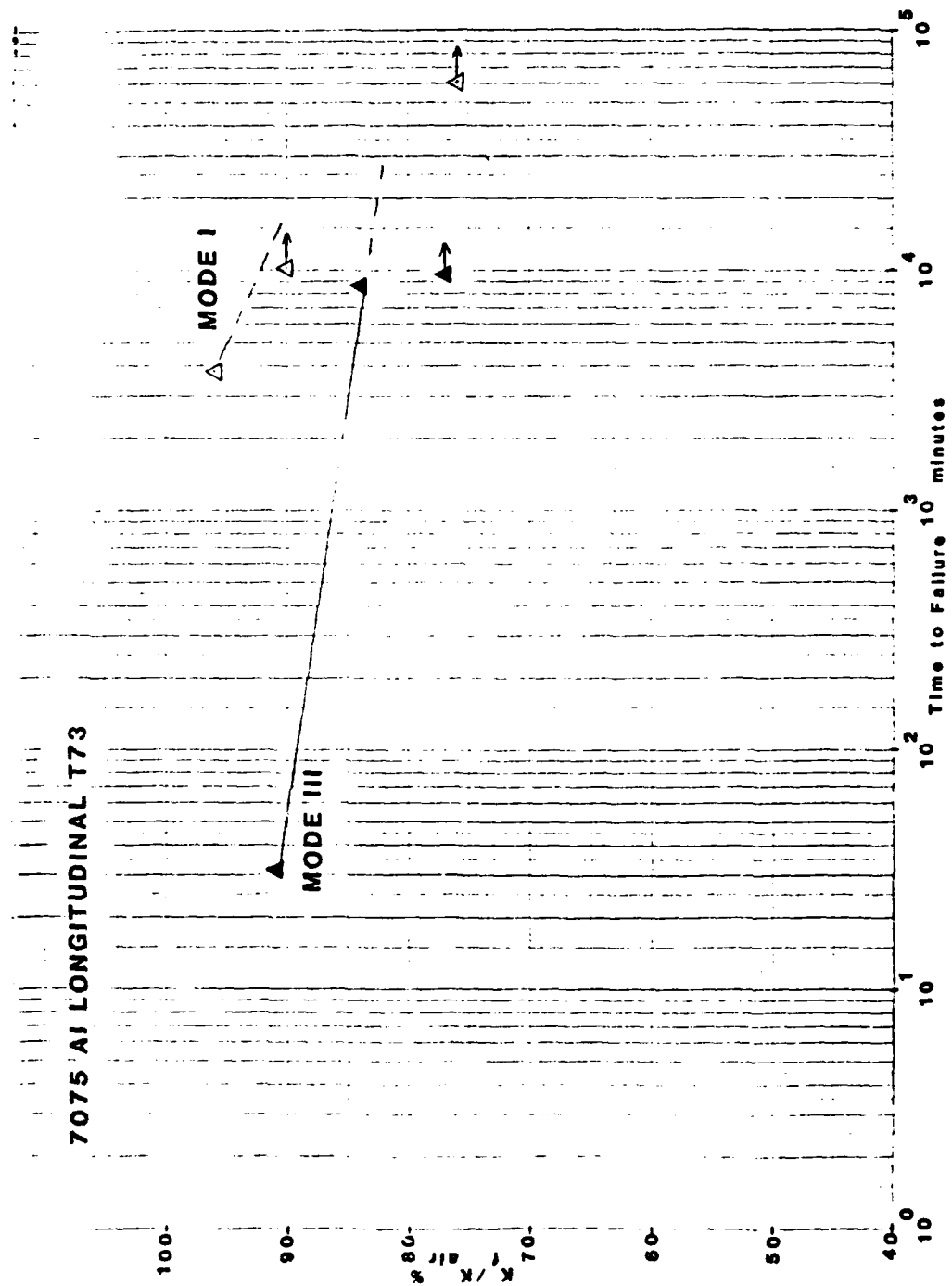


Figure 11 Constant Load/Torque SCC Results for Overaged 7075 Al - Longitudinal

7075 Al ST
MODE I SUMMARY

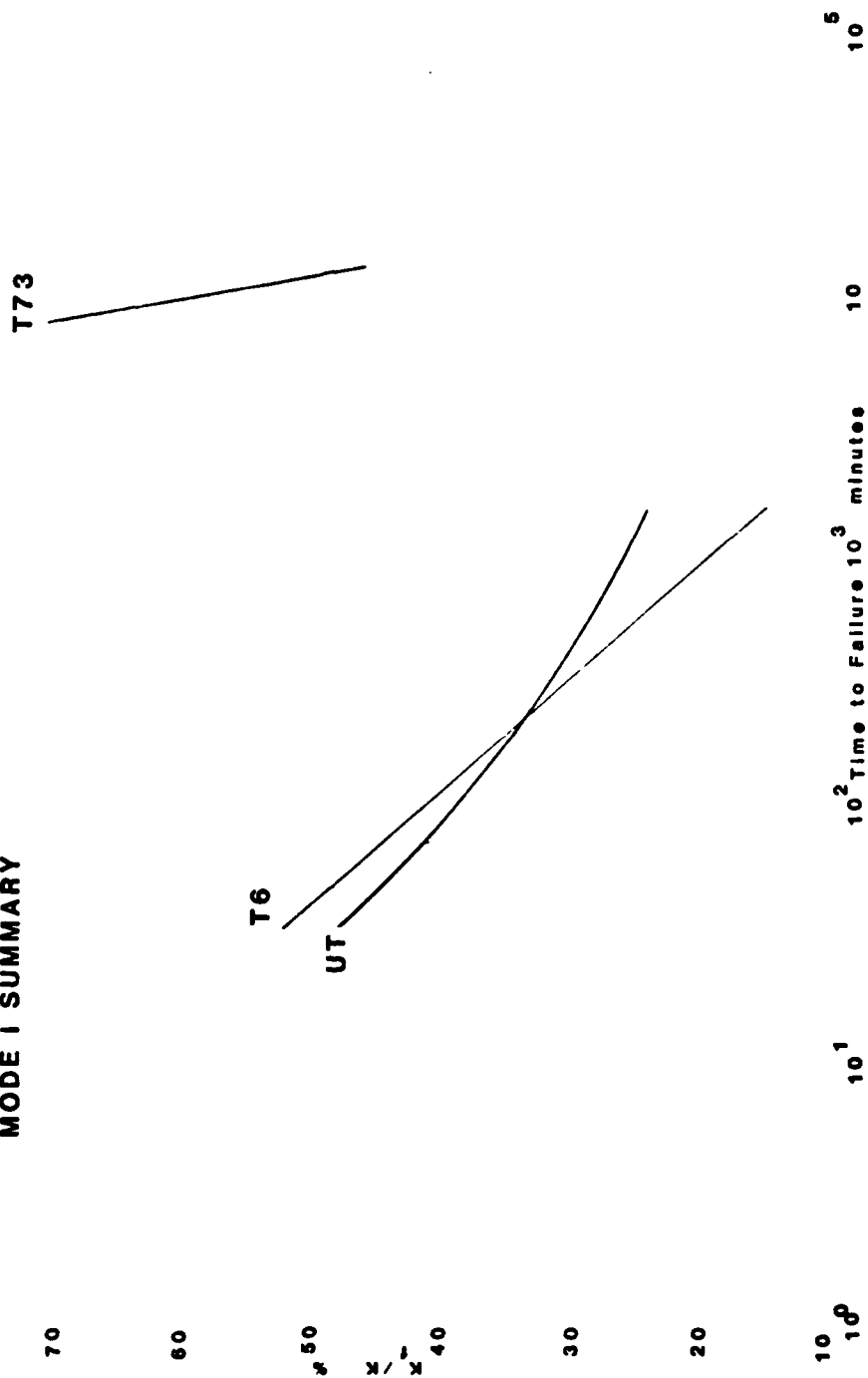


Figure 12 Constant Load SCC Summary for 7075 Al - ST Orientation

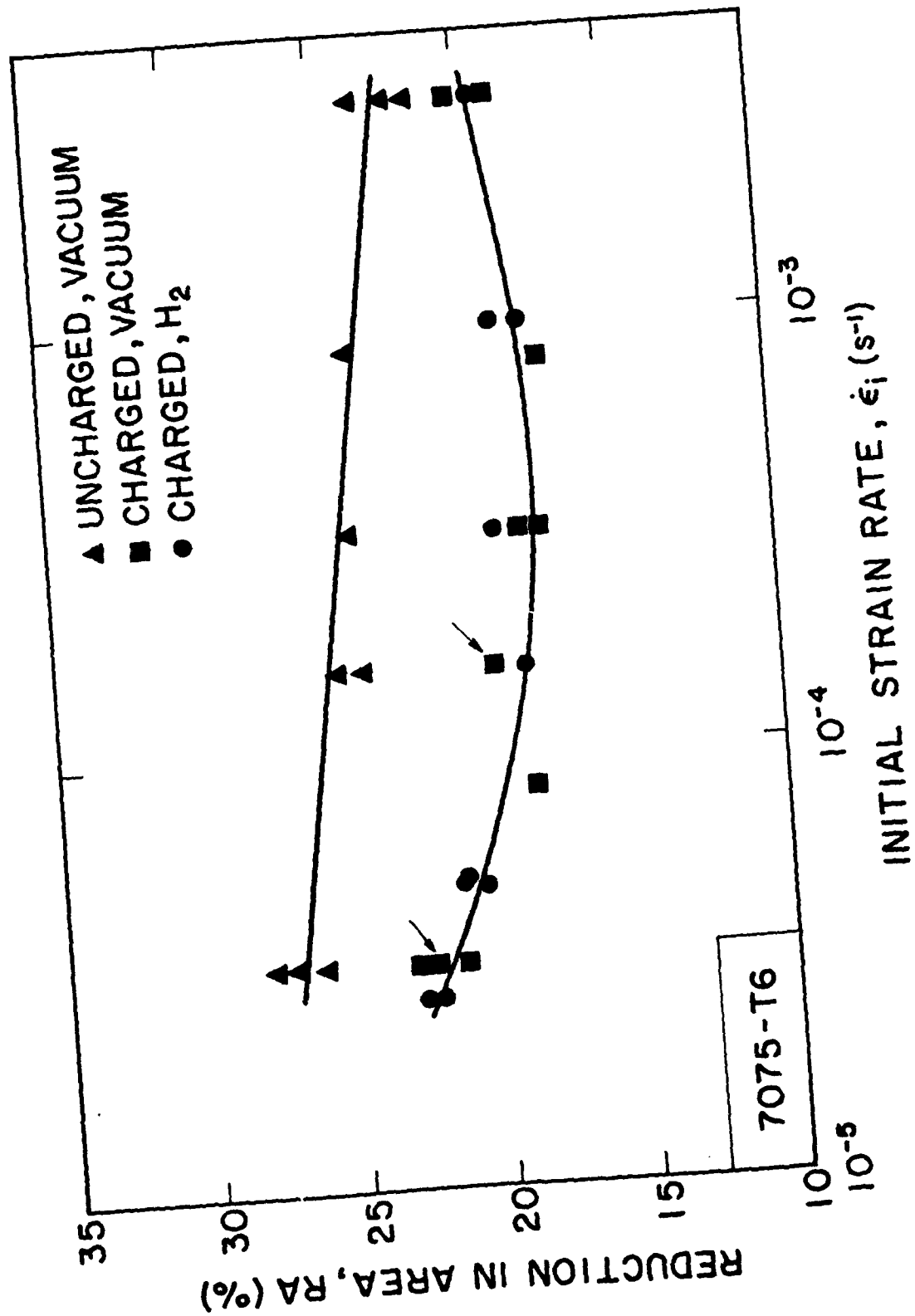


Figure 13 Ductility (RA) as a Function of Initial Strain Rate for Al 7075-T6 Specimens

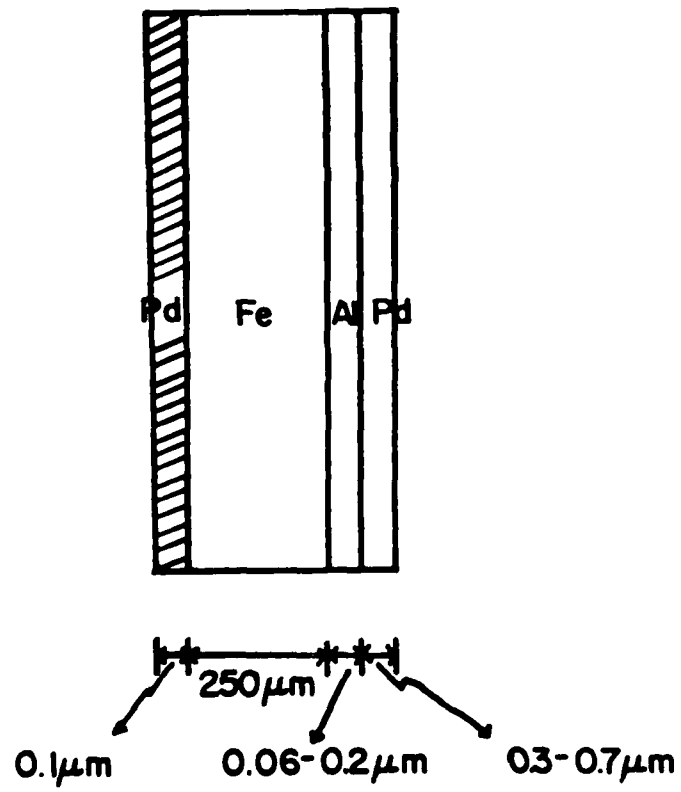


Figure 14 Composite Specimen for Measuring Hydrogen Diffusivity in Aluminum

FILM
5-8